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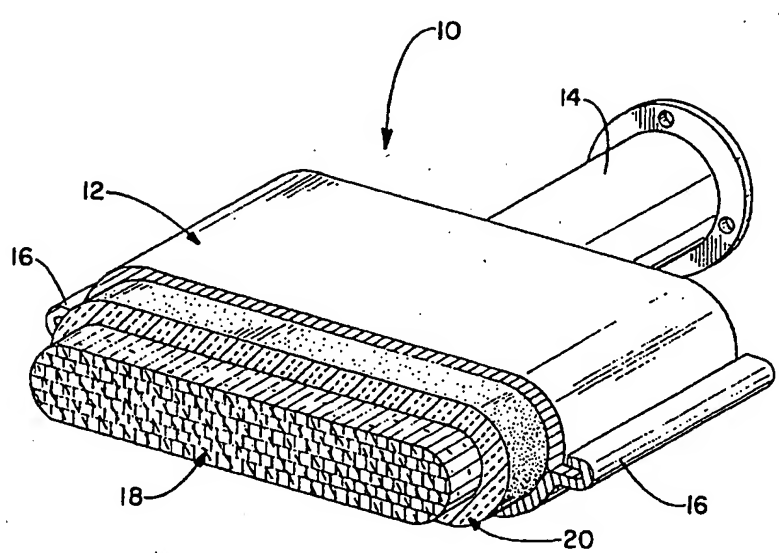


(57) Abstract

A mounting mat (20) suitable for catalytic converters (10), or diesel particulate traps comprising an integral composite sheet comprising ceramic fibers and a binder, wherein the fibers are substantially shot free and have an average length in the range of about 1 cm to about 10 cm. The composite sheet has a nominal thickness of about 3 mm to about 30 mm, a nominal density of about 0.03 to about 30 mm grams per cubic centimeter and the mat (20) has flexible, structural integrity. The mounting mat (20) is capable of exerting stable pressure under fixed gap conditions over an operating temperature range of about 20 °C to about 1200 °C. A method of mounting a fragile structure in a device is also provided.

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# MOUNTING MAT FOR FRAGILE STRUCTURES SUCH AS CATALYTIC CONVERTERS

## Field of the Invention

The present invention is directed to a mounting mat for a fragile structure such as a catalytic converter or diesel particulate trap. More particularly, it is directed to a mounting mat having enhanced handleability characteristics comprising a composite mat of ceramic fiber for mounting and supporting frangible material.

## Background of the Invention

Catalytic converter assemblies for treating exhaust gases of automotive and diesel engines contain a catalyst support structure for holding the catalyst, used to effect the oxidation of carbon monoxide and hydrocarbons and the reduction of oxides of nitrogen, the support structure being mounted within a metal housing. The support structure is generally made of a frangible material, such as a monolithic structure formed of metal or a brittle, fireproof ceramic material such as aluminum oxide, silicon dioxide, magnesium oxide, zirconia, cordierite, silicon carbide and the like. These materials provide a skeleton type of structure with a plurality of tiny flow channels. Small shockloads or stresses are sufficient to crack or crush the monolith.

The support structure is contained within a metal housing, with a space or gap between the external surface of the support structure and the internal surface of the housing. At least one sheet of mounting material is positioned within the gap between the support structure and

the housing, to provide thermal insulation and to protect the support structure from both thermal and mechanical shock. Examples of catalytic converter devices in which a fragile structure is mounted are contained in U.S. Patent Nos. 4,863,700; 4,999,168; and 5,032,441, which are incorporated herein by reference.

Conventional materials exhibit difficulties when the catalytic converter operating temperature is either very low (20-300°C) or very high (750-1200°C and above). Conventional mounting materials have exhibited failure in catalytic converters used in vehicles having a higher gross weight than normal gasoline powered passenger automobiles.

Because of their high gross vehicle weight, the engines of such vehicles operate at a much higher percentage of their maximum output for a much greater percentage of their operating time, than do the engines in passenger automobiles. These operating conditions in heavier vehicles result in maximum catalytic converter temperatures of much greater than 850°C. Converter monolith temperatures of 1050°C are not uncommon and temperatures in excess of 1200°C may be encountered.

A typical passenger automobile catalytic converter utilizes a ceramic monolith which is supported by an intumescent mounting material having a nominal thickness of about 4.95 mm to about 9.9 mm and a nominal density of about 0.63g/cm$^3$. This material is compressed during installation of the ceramic monolith into its metallic housing to a nominal thickness of about 3.1 mm to about 6.2 mm and a nominal density of about 1 g/cm$^3$. The conventional intumescent material contains vermiculite which expands at about 300°C and degrades at temperatures greater than 750°C.

It is desirable to maintain a constant pressure on the metallic housing and the catalyst support structure under all conditions. However, upon the initial heating of the catalytic converter assembly, particularly the initial cycles, conventional mounting materials experience a tremendous expansion pressure which can crush the catalyst support structure and cause component failure.

Conventional intumescent material meets the needs of most current automotive converters, but does not meet the needs of several near future requirements as well as some current diesel and heavy duty truck requirements. These requirements are focused upon the maintenance of near constant residual mounting pressure in temperature regimes below 300°C and above 750°C. Examples of severe condition applications in which these properties are important include the following: 1) Close-coupled converters which are mounted closer to the engine for better conversion efficiency via higher gas temperatures (about 750°C); 2) Diesel convertors and diesel particulate traps which operate at low temperatures and which are commonly pre-heated at 500°C to pre-expand the intumescent mat prior to installation in the vehicle. This "pre-heating" would be unnecessary with the mounting mat of the present invention. 3) Heavy-duty truck converters and motorcycle converters which run at temperatures which greatly exceed 750°C. 4) Thin wall monoliths which will assist in meeting future EPA requirements via reaching operating temperature quicker due to their lighter mass. These monoliths are weak and will be crushed by the dramatic pressure increase of intumescent mats.

Intumescent mat would fail in the above cited severe condition application examples due to lack of expansion at low temperatures, to high pressure excursions between 300-750°C, and to loss of pressure above 750°C. With lack of expansion or loss of pressure the fragile monolith would be released, rattle about within the can, and self-destruct due to mechanical shock. With high pressure excursions, low strength monoliths would be crushed.

Alternative mounting materials have been investigated for severe condition applications, however, many have been found to be difficult and cumbersome to handle and to fabricate into catalytic converter assemblies. The mounting materials proposed to accommodate the severe condition applications are themselves fragile, and require expensive preprocessing such as stitchbinding prior to installation, and may require combination with other mounting materials, such as intumescent sheets and backing layers. These mounting materials are generally very thick and lack structural integrity, even being handled in a bag to prevent crumbling. Thus they are difficult to cut to size for installation, and further must be compressed substantially to fit enough material needed for supportive mounting within the gap between the catalyst support structure and the housing. Consequently, "flashing" commonly occurs, with excess material being squeezed out of the housing. Examples of such alternate approaches are found in US Patent Nos 4,693,338; 4,929,429 and 5,028,397.

Diesel particulate traps similarly include one or more porous tubular or honeycomb-like structures (having channels closed at one end, however) which are mounted by a thermally resistant material within a housing.

Particulate is collected from exhaust gases in the porous structure until regenerated by a high temperature burnout procedure, which thermally taxes the mounting material.

## Summary of the Invention

It is an object of the present invention to provide a mounting mat possessing good handleability and fabrication characteristics capable of withstanding high temperatures without degradation while maintaining stable pressure over a wide range of operating temperatures.

The present invention provides a mounting mat comprising an integral composite sheet comprising ceramic fibers and a binder, wherein said fibers are substantially shot free and have an average length in the range of about 1 cm to about 10 cm, and wherein said mat has flexible structural integrity and is capable of exerting substantially constant pressure under fixed gap conditions over an operating temperature range of about 20°C to about 1200°C. Said composite sheet may have a thickness of about 3 mm to about 30 mm, and a nominal density of about 0.03 to about 0.3 grams per cubic centimeter.

The present invention further provides a device for treatment of exhaust gases comprising:

(a) a housing having an inlet at one end and an outlet at its opposite end through which exhaust gases flow;

(b) a structure resiliently mounted within said housing, said structure having an outer surface and an inlet end face at one end in communication with said inlet of said housing and an outlet end face at its opposite end in communication with said outlet of said housing;

(c) a flexible mounting mat in contact with and covering at least a portion of said outer surface of said

structure; and disposed between said structure and said housing, wherein said mounting mat comprises an integral composite sheet of ceramic fibers and a binder, wherein said fibers are substantially shot free and have an average length in the range of about 1 cm to about 10 cm, and said sheet is capable of exerting substantially stable mounting pressure over a temperature range of about 20°C to about 1200°C.

The mounting mat of the present invention may be used to mount any fragile or frangible structure, such as an automotive catalytic converter catalyst support monolith or diesel particulate trap, and the like, in all expected temperature environments where protection from thermal and mechanical shock is desirable. The mounting mat of the present invention maintains a near constant pressure over the entire operating range of all current and known future converter/trap designs. Throughout this Specification, references to catalytic converters should be considered generally to apply to diesel particulate traps.

A method is provided by the present invention of mounting a fragile structure having at least one inlet face within a device having a housing to provide thermal insulation and mechanical shock resistance comprising: wrapping a flexible mounting mat comprising an integral composite sheet of ceramic fibers and binder around the entire perimeter of at least a portion of the structure's surfaces adjacent to the inlet face, and forming a housing around the wrapped structure and radially compressing the mounting mat between the structure and the housing, wherein the fibers are substantially shot free and have an average length in the range of about 1 cm to about 10 cm and wherein said mat is capable of exerting substantially

stable pressure over an operating temperature range of about 20°C to about 1200°C. For use in catalytic converters or diesel particulate traps, said composite sheet has an uninstalled nominal thickness of about 3 mm to about 30 mm, an uninstalled nominal density of about 0.03 to about 0.3 grams per cubic centimeter, and an installed thickness of about 2 mm to about 8 mm and a gap bulk density of about 0.1 to about 1.5 grams per cubic centimeter.

## Brief Description of the Drawings

Fig. 1 is a fragmentary, elevational view of a catalytic converter according to the present invention.

Fig. 2 is a graphical representation of pressure versus temperature for mounting mats of the present invention compared to conventional converter mats at various gap bulk densities.

Fig. 3 is a graphical representation of pressure versus temperature for mounting mats of the present invention at various gap bulk densities.

## Detailed Description of the Invention

The mounting mat of the present invention possesses good handleability and is easily processed in the fabrication of devices utilizing its capabilities of maintaining substantially stable pressure under compression in fixed gap conditions over a wide range of operating temperatures. The mounting mat provides a resilient means of protecting fragile structures from mechanical shock.

Referring to the Figures, there is shown in Fig. 1 a catalytic converter 10 generally. The present invention is not intended to be limited to use in the catalytic

converter shown, and so the shape is shown only as an example to illustrate the invention. In fact, the mounting mat could be used to mount any fragile structure, such as a diesel particulate trap or the like. Nonautomotive applications for the mounting mat of the present invention include but are not limited to catalytic converters for chemical industry emission (exhaust) stacks. The term fragile structure is intended to mean and include structures such as metal or ceramic monoliths or the like which are fragile or frangible in nature and would benefit from a mounting mat such as is described herein.

Catalytic converter 10 includes a generally tubular housing 12 formed of two pieces of metal, e.g. high temperature-resistant steel. Housing 12 includes an inlet 14 at one end and an outlet (not shown) at its opposite end. The inlet 14 and outlet are suitably formed at their outer ends whereby they may be secured to conduits in the exhaust system of an internal combustion engine. Device 10 contains a fragile catalyst support structure, such as a frangible ceramic monolith 18 which is supported and restrained within housing 12 by mounting mat 20, to be further described. Monolith 18 includes a plurality of gas-pervious passages which extend axially from its inlet end face at one end to its outlet end face at its opposite end. Monolith 18 is constructed of a suitable refractory metal or ceramic material in known manner and configuration. Monoliths are typically oval or round in cross-sectional configuration, but other shapes are possible.

In accordance with the present invention, the monolith is spaced from its housing by a distance or a gap, which will vary according to the type and design of converter or

trap utilized. This gap is filled with mounting mat 20 to support the ceramic monolith 18. The mounting mat 20 provides both thermal insulation to the external environment and mechanical support to the catalyst support structure.

The mounting mat comprises an integral composite sheet of ceramic fibers and a binder. By integral is meant that after manufacture the mounting mat has self supporting structure, needing no reinforcing or containment layers of fabric, plastic or paper, and can be handled or manipulated without disintegration.

Ceramic fibers which are useful in the mounting mat of the present invention include polycrystalline oxide ceramic fibers such as mullite, alumina, high alumina aluminosilicates, aluminosilicates, zirconia, titania, chromium oxide and the like. The ceramic fibers are preferably refractory. When the ceramic fiber is an aluminosilicate, the fiber may contain between about 55 to about 98% alumina and between about 2 to about 45 % silica, with the preferred ratio of alumina to silica being between 70 to 30 and 75 to 25. Suitable polycrystalline oxide refractory ceramic fibers and methods for producing the same are contained in US Patent Nos. 4,159,205 and 4,277,269, which are incorporated herein by reference. FIBERMAX® polycrystalline mullite ceramic fibers are available from The Carborundum Company, Niagara Falls, New York in blanket, mat or paper form.

The diameters of fibers useful in the present invention are generally about 1 micron to about 10 microns, and the length of the fibers are generally about 1 cm to about 10 cm, preferably about 1.25 cm to about 7.75 cm. The fibers used in the present invention are characterized

by being substantially shot free, having very low shot content, generally on the order of about 5 percent nominally or less.

The ceramic fibers are processed into a mat by conventional means such as dry air laying. The mat at this stage, has very little structural integrity and is very thick relative to the conventional catalytic converter and diesel trap mounting mats.

The mat is further processed by the addition of a binder to the mat by impregnation to form a discontinuous fiber composite. The binder is added after formation of the mat, rather than forming the mat prepreg by introduction and dispersion of fibers into water or a binder slurry, in order to maintain fiber length by reducing breakage.

Suitable binders include aqueous and nonaqueous binders, but preferably the binder utilized is a reactive, thermally setting latex which after cure is a flexible material that can be burned out of the installed mounting mat. Examples of suitable binders or resins include, but are not limited to, aqueous based latexes of acrylics, styrene-butadiene, vinylpyridine, acrylonitrile, vinyl chloride, polyurethane and the like. Other resins include low temperature, flexible thermosetting resins such as unsaturated polyesters, epoxy resins and polyvinyl esters. Specific binders useful in the present invention include but are not limited to HI-STRETCH V-60™, a trademark of B.F. Goodrich Co. (Akron, Ohio) for acrylonitrile based latex. Solvents for the binders can include water, or a suitable organic solvent, such as acetone, for the binder utilized. Solution strength of the binder in the solvent (if used) can be determined by conventional methods based

on the binder loading desired and the workabilitiy of the binder system (viscosity, solids content, etc.).

Methods of impregnation of the mat with the binder include complete submersion of the mat in a liquid binder system, or alternatively spraying the mat. In a continuous procedure, a ceramic fiber mat which can be transported in roll form, is unwound and moved, such as on a conveyer or scrim, past spray nozzles which apply the binder to the mat. Alternatively, the mat can be gravity-fed past the spray nozzles. The mat/binder prepreg is then passed between press rolls which remove excess liquid and densify the prepreg to approximately its desired thickness.

The densified prepreg is then passed through an oven to remove any remaining solvent and if necessary to partially cure the binder to form a composite. The drying and curing temperature is primarily dependent upon the binder and solvent (if any) used. The composite can then either be cut or rolled for storage or transportation.

The mounting mat can also be made in a batch mode, by immersing a section of the mat in a liquid binder, removing the prepreg and pressing to remove excess liquid, thereafter drying to form the composite and storing or cutting to size.

The resin loading level in the composite is generally on the order of about 0.5% to about 20%, and preferably is about 2% to about 7%. The compressed, bonded composite is flexible and has structural integrity and good handleability.

The composite can be cut, such as by die stamping, to form mounting mat of exact shapes and sizes with reproducible tolerances. The composite mounting mat may be bent back upon itself without cracking, due to its

flexibility. This mounting mat 20 can be easily and flexibly fitted around the catalyst support structure 18 without cracking and fabricated into the catalytic converter housing 12 to form a resilient support for the catalyst support structure 18, with minimal or no flashing such as by extrusion or flow of excess material into the flange 16 area. The handleability and processability of the mounting mat 20 will permit the fabrication of the catalytic converter assembly 10 to be substantially automated.

EXAMPLE

A mounting mat composite was prepared in a batch mode by placing a 12 inch by 36 inch (30 cm by 91 cm) mat of FIBERMAX® polycrystalline ceramic fibers on a wax paper covered sheet of plexiglass in a container and pouring onto the mat a 3% solution of HI STRETCH V60™ acrylonitrile based latex in water, in an amount calculated to give a loading of 6.5% organics in the composite. An aluminum screen was pressed on top of the mat to extract excess binder solution, and was removed. Wax paper followed by plexiglass was placed over the mat to form a sandwich orientation, and the assembly was pressed in a Williams paper press to a thickness of 3/16 inch (about 0.5 cm). The glass and wax paper were removed and the impregnated mat was placed on a mold release-treated aluminum foil in an oven to dry at 145-150°C for 45 to 60 minutes. The dried composite was strong, flexible and easy to handle.

The mounting mat of the present invention generally has a nominal thickness (before compression during device assembly) of about 3 mm to about 30 mm. The nominal density, being the calculated density of the mounting mat without being compressed, is generally about 0.03 to about

0.3 grams per cubic centimeter.

When the mounting mat 20 is placed into the catalytic converter 10 during fabrication of the device, the mounting mat is radially compressed between the members of the housing 12 to a thickness corresponding to the gap between the housing 12 and the catalyst support structure 18, generally about 2 mm to about 8 mm, preferably about 2 mm to about 6 mm. This increases the density of the mounting mat, to its final gap bulk density, and results in the mounting mat exerting pressure under operating conditions against the adjacent elements 12 and 18. Depending upon the application, the mounting mats of the present invention can exert stable mounting pressures from about 0.1 $Kg/cm^2$ to about 50 $kg/cm^2$.

In operation, the catalytic converter experiences a significant change in temperature. Due to the differences in their thermal expansion coefficients, the housing 12 may expand more than the support structure 18, such that the gap between these elements will increase slightly. The thickness of mounting mat 20 is selected such that even at operating temperatures the gap is filled with mounting mat material, although at a slightly lower pressure than at ambient temperatures, to prevent the support structure 18 from vibrating loose. The substantially stable mounting pressure exerted by the mounting mat 20 under these conditions permits accommodation of the thermal characteristics of the assembly without compromising the physical integrity of the constituent elements.

Conventional intumescent mats may experience an increase of pressure of up to 800% upon heating to operating temperatures under standard test fixed gap

conditions. Even in expanding gap conditions of normal operation, these conventional mats may crack fragile catalyst support structures. The mounting mat of the present invention maintains substantially stable mounting pressure under standard test fixed gap conditions, and may experience a slight decrease in pressure of up to about 30% in strenuous operating, expanding gap conditions at a given bulk density. The selection of bulk density for mounting mat 20 in a given application will maintain the necessary protective mounting pressure on the housing 12 and support structure 18.

Fixed gap pressure measurements are carried out in an enclosed furnace chamber having a roof orifice and a floor orifice. A pair of fused quartz rams are placed in the furnace, one per orifice. The quartz rams are of sufficient length to extend from the furnace's center to a distance beyond the furnace's exterior shell. At the center of the furnace the two quartz ram ends form the "fixed gap" between which the sample is placed. Outside the furnace the extending rams are rigidly mounted to load cells. The sample's pressure characteristics, in a specified fixed gap condition, are monitored by these load cells as the furnace is ramped upwards in temperature.

Fig. 2 is a graph showing pressure of mounting mats according to the present invention as compared to intumescent papers at various gap bulk densities under fixed gap conditions. It is demonstrated that the inventive mounting mats exert a stable, substantially constant mounting pressure over a wide temperature range as compared to conventional intumescent papers.

Fig. 3 is a graph showing pressure of mounting mats according to the present invention over a range of

temperatures under fixed gap conditions. Again, stable, substantially constant mounting pressure throughout the temperature range is demonstrated.

The dramatic expansion pressure increase during the initial thermal cycles observed using conventional catalytic converter mounting materials are not observed using the mounting mat of the present invention.

A mounting mat according to the present invention was prepared from FIBERMAX® polycrystalline ceramic fibers and tested for hot gas erosion resistance using the traditional procedure used to measure conventional converter mats. The test conditions were maintaining an oven temperature of 600°C while pulsing air 2.0 seconds on and 0.5 seconds off, at an air velocity of 300 meters per second. Conventional intumescent paper mats eroded 2.54 cm in 2 to 82 hours, while the mounting mat of the present invention showed no erosion in 100 hours of testing.

The superior physical property characteristics demonstrated by the mounting mats of the present invention over conventional converter/diesel trap mats, such as high erosion resistance and substantially constant, stable pressure over a wide temperature range, are desirable in both catalytic converter and diesel trap designs. The mounting mats can be die cut and are additionally operable as resilient supports in a thin profile, providing ease of handling, and in a flexible form, so as to be able to provide a total wrap of the catalyst support structure without cracking. Alternatively, the mounting mat may be integrally wrapped about the entire circumference or perimeter of at least a portion of the catalyst support structure. The mounting mat may eliminate the need for an end-seal currently used in conventional converter devices

to prevent gas by-pass.

The mounting mat of the present invention is useful in applications such as catalytic converters or diesel particulate traps which utilize low strength monoliths and/or experience either unconventionally low operating temperatures (less than about 300°C) or high operating temperatures (above about 750°C), as well as traditional mounting mat applications which currently use difficult to handle containment/fiber blanket forms.

The mounting mat of the present invention can also be used in catalytic converters employed the chemical industry which are located within exhaust or emission stacks, and which also contain fragile honeycomb type structures to be protectively mounted.

Thus, the objects of the invention are accomplished by the present invention, which is not limited to the specific embodiments described above, but which includes variations, modifications and equivalent embodiments defined by the following claims.

We claim:

1. A mounting mat comprising an integral composite sheet comprising ceramic fibers and a binder, wherein said fibers are substantially shot free and have an average length in the range of about 1 cm to about 10 cm, and wherein said mat has flexible structural integrity and is capable of exerting substantially constant pressure under fixed gap conditions over an operating temperature range of about 20°C to about 1200°C.

2. The mounting mat of claim 1 wherein said composite sheet has a thickness of about 3 mm to about 30 mm, and a nominal density of about 0.03 to about 0.3 grams per cubic centimeter.

3. The mounting mat of claim 1 wherein said fibers have diameters in the range of about 1 micron to about 10 microns.

4. The mounting mat of claim 1 wherein said fibers have less than about 5% shot.

5. The mounting mat of claim 1 wherein said fibers are selected from the group consisting of alumina, mullite, high alumina aluminosilicates, zirconia, titania, chromium oxide and mixtures thereof.

6. The mounting mat of claim 1 wherein said fibers are mullite.

7. The mounting mat of claim 1 wherein said fibers are aluminosilicate comprising about 55% to about 98% alumina and about 2% to about 45% silica.

8. The mounting mat of claim 1 wherein said binder is selected from the group consisting of latexes of acrylics, styrene-butadiene, vinylpyridine, acrylonitrile, vinyl chloride and polyurethane.

9. The mounting mat of claim 1 wherein said binder is a flexible thermosetting resin selected from the group consisting of unsaturated polyesters, epoxies and polyvinyl esters.

10. The mounting mat of claim 1 wherein the loading of binder in said composite sheet is about 0.5% to about 20%.

11. A device for the treatment of exhaust gases comprising;

(a) a housing having an inlet at one end and an outlet at its opposite end through which exhaust gases flow;

(b) a structure resiliently mounted within said housing, said structure having an outer surface and an inlet end face at one end in communication with said inlet of said housing and an outlet end face at its opposite end in communication with said outlet of said housing;

(c) a flexible mounting mat in contact with and covering at least a portion of said outer surface of said structure; and disposed between said structure and said housing, wherein said mounting mat comprises an integral composite sheet of ceramic fibers and a binder, wherein said fibers are substantially shot free and have an average

length in the range of about 1 cm to about 10 cm, and said sheet is capable of exerting substantially stable mounting pressure over a temperature range of about 20°C to about 1200°C.

12. The device as in claim 11 wherein said flexible mounting mat is integrally wrapped about the entire perimeter of at least a portion of said structure.

13. The device of claim 11 wherein said fibers are selected from the group consisting of alumina, mullite, high alumina aluminosilicates, aluminosilicates, zirconia, titania, chromium oxide and mixtures thereof.

14. The device of claim 11 wherein said fibers are aluminosilicate comprising about 55% to about 98% alumina and about 2% to about 45% silica.

15. The device of claim 11 wherein said mounting mat is compressed to an installed thickness of about 2 mm to about 8 mm.

16. The device of claim 11 wherein the mounting pressure is between about 0.1 $kg/cm^2$ and about 50 $kg/cm^2$.

17. A method of mounting a fragile structure having at least one inlet face within a device having a housing to provide thermal insulation and mechanical shock resistance comprising the steps of:

a) wrapping a flexible mounting mat comprising an integral composite sheet of refractory ceramic fibers and a binder around the entire perimeter of at least a portion

of the structure's surfaces adjacent to the inlet face, and

b) forming a housing around the wrapped structure, and radially compressing said mounting mat between said structure and said housing,

wherein said fibers are substantially shot free and have an average length in the range of about 1 cm to about 10 cm,

and wherein said mat is capable of exerting substantially stable expansion pressure over an operating temperature range of about 20°C to about 1200°C.

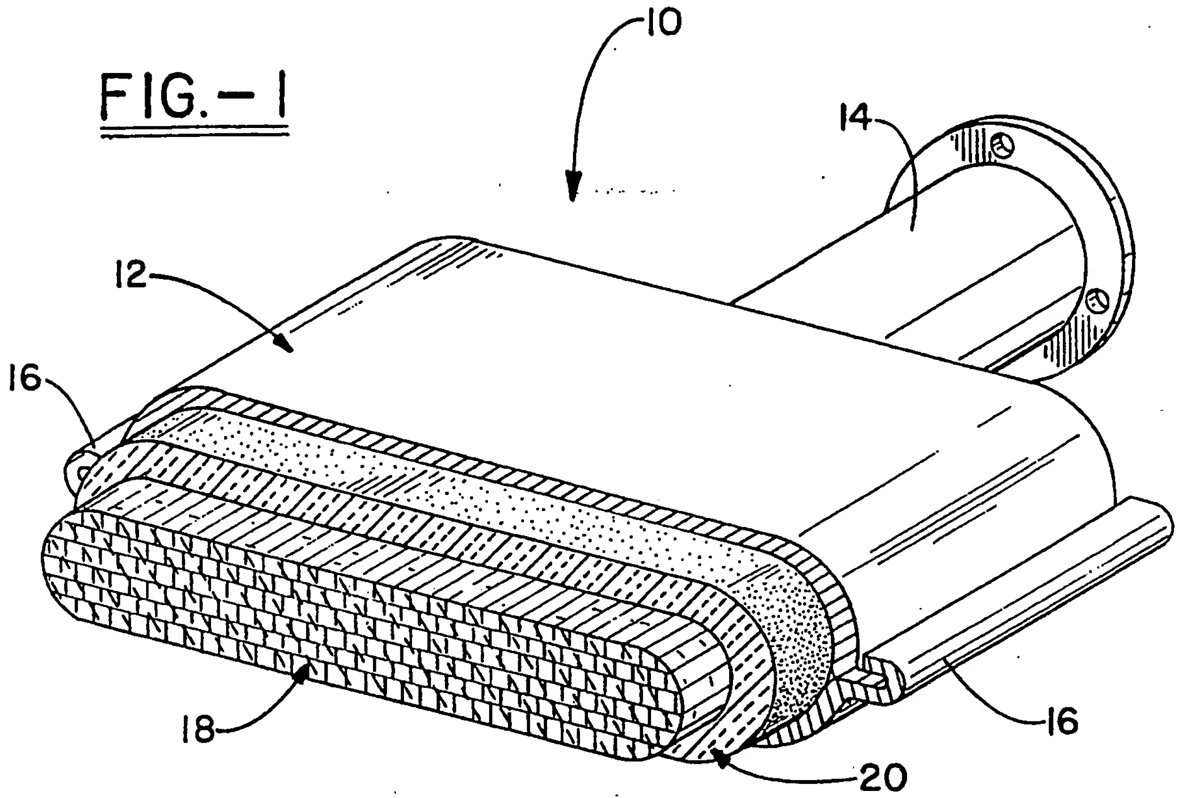
18. The method as in claim 17 wherein said composite sheet has an uninstalled nominal thickness of about 3 mm to about 30 mm, an uninstalled nominal density of about 0.03 to about 0.3 grams per cubic centimeter, and an installed thickness of about 2 mm to about 8 mm.

19. The method as in claim 18 wherein said device is a catalytic converter.

20. The method as in claim 18 wherein said device is a diesel particulate trap.

21. The method as in claim 17 wherein said device is an emission exhaust stack catalytic converter.

FIG.-1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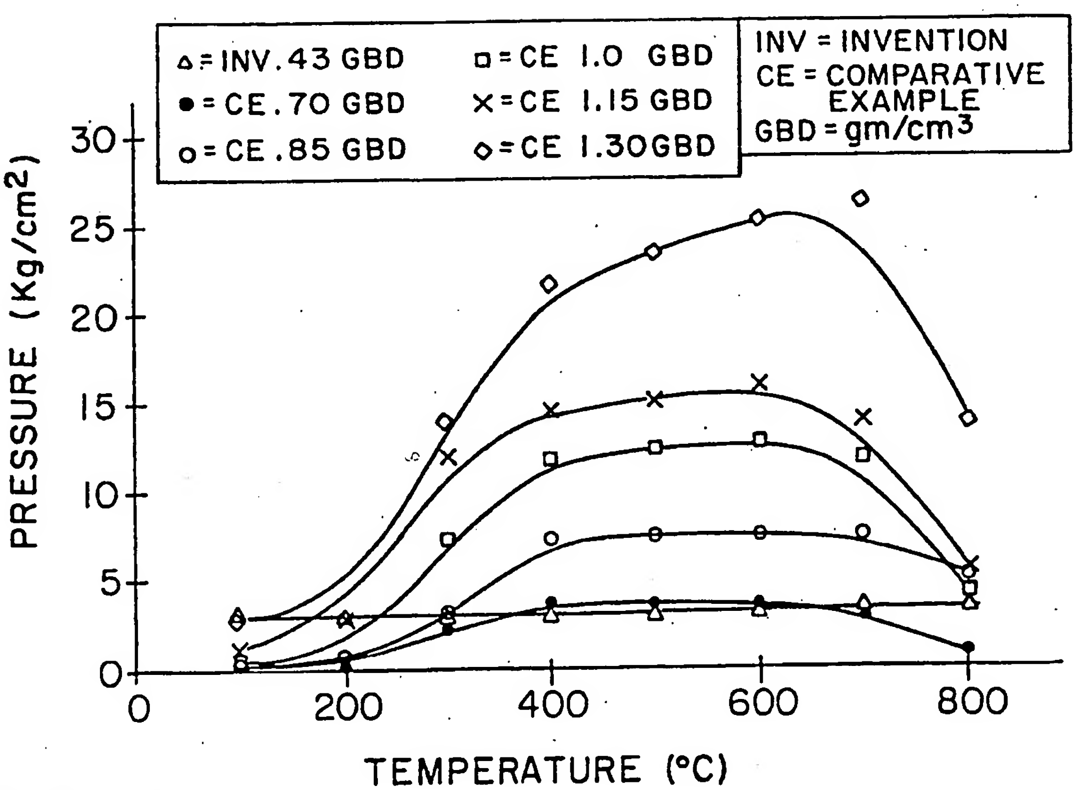
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CE = COMPARATIVE EXAMPLE
GBD = gm/cm3
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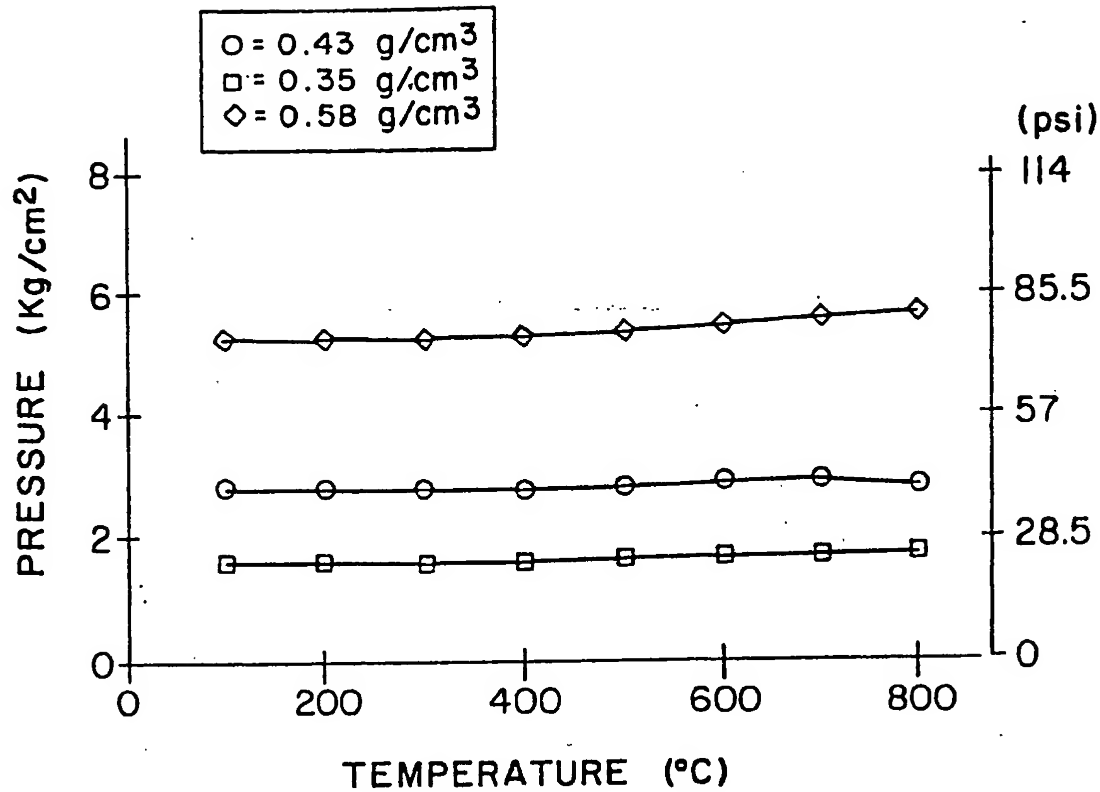
TEMPERATURE (°C)

FIG.-2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2
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0
200
400
600
800
TEMPERATURE (°C)

FIG.-3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/04405

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(5) :F01N 3/28
US CL :422/179, 180, 221, 222; 501/95; 252/62; 428/114
According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 422/179, 180, 221, 222; 501/95; 252/62; 428/114; 60/299, 301

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

NONE

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| Y | US, A, 4,929,429 (MERRY) 29 MAY 1990, SEE ENTIRE DOCUMENT. | 1-21 |
| Y | US, A, 3,771,967 (NOWAK) 13 NOVEMBER 1973, SEE ENTIRE DOCUMENT. | 1-21 |
| Y | US, A, 5,032,441 (TEN EYCK ET AL) 16 JULY 1991, SEE ENTIRE DUCUMENT. | 1-21 |
| Y | US, A, 4,752,515 (HOSOI ET AL) 21 JUNE 1988, SEE ENTIRE DOCUMENT. | 7, 14 |
| Y | US, A, 3,795,524 (SOWMAN) 05 MARCH 1974, SEE ENTIRE DOCUMENT. | 9 |
| Y | US, A, 3,996,145 (HEPBURN) 07 DECEMBER 1976, SEE ENTIRE DOCUMENT. | 9 |

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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|---|---|
| Date of the actual completion of the international search<br>23 MAY 1994 | Date of mailing of the international search report<br>JUN 24 1994 |
| Name and mailing address of the ISA/US<br>Commissioner of Patents and Trademarks<br>Box PCT<br>Washington, D.C. 20231<br>Facsimile No. (703) 305-3230 | Authorized officer<br>HIEN TRAN Jill Warden for<br>Telephone No. (703) 308-0196 |

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